Frequently Asked Questions Concerning Standard Methods and 40 CFR Part 136
Revision 1 Sept. 2007

These are questions prompted by the recent major changes to 40 CFR Part 136 concerning approved methods for the analysis of wastewater. Included are questions concerning quality control requirements found in *Standard Methods*. Interpretation of these changes and methods is an ongoing process for both EPA and VA DEQ. Your questions often prompt discussions that may alter the agencies' original interpretations or intent. Questions 1-5 clarify information provided during the recent training on changes to 40 CFR. Changes to the original document are in red font.

1. Why is EPA requiring bacteriological samples to be analyzed within 2 hrs of receipt at the laboratory (Table II, footnote 22) instead of the 6 hr holding time we had before?
    a. EPA now indicates that as long as the bacteriological sample is maintained at <10 degrees C, the holding time will be a maximum of 6 hours with set-up completed within 2 hours. This means that the sample must be logged in at the lab within 6 hours of collection. EPA still prefers that sample analysis begin within 2 hours of collection.

2. As stated in *Standard Methods* 2550B, will DEQ require etched-glass thermometers with 0.1-degree markings for all temperature readings?
    a. No. If the type of thermometer in use prior to March 12, 2007 was acceptable for compliance monitoring, use of that type may continue.

3. Is the acceptance range for pH 90-110% when determining Certification of Operator Competence and 80-120% for Initial Demonstration of Capability (IDC)?
    a. No. Use +/- 0.1 S.U. of the true value of the buffer being tested. (This is the same acceptance range for checking pH calibration.)

4. For which methods will DEQ require the lab to determine Method Detection Limits (MDL)?
    a. For DEQ's Wastewater – None. Check the reagent blanks (RB) [See # 9a] by comparing them to the Minimum Quantitation Level (MQL) the lab has determined for each method. The concentration of a reagent blank is acceptable if it is below the MQL. [See # 10] **Note**: this is a revision of

information provided during the some of the training sessions on changes to 40 CFR Part 136.

5. Must I collect two 24-hr composite samples each day so that I have duplicate samples for analysis?
    a. No. Pour two aliquots directly from the compositor container in order to create duplicate samples. (Do not pour one aliquot and then divide that into two separate samples.) When doing duplicate samples you are demonstrating that the entire composite sample was properly mixed prior to removing aliquots for analysis.

       Standard Methods defines '**duplicates**' as "two samples taken at the same time at the same location." The purpose of duplicates is to determine the precision of the analyst and/or method and the homogeneity of the sample. (Is the analyst, method, and/or sampling capable of obtaining repeatable results?)

6. My facility has three outfalls. Must I run a duplicate each day of testing for each outfall?
    a. No. *Standard Methods* requires a duplicate each day of analysis or on 5% of samples analyzed. This requirement is NOT dependent on the outfall or sampling site but rather on the total number of samples analyzed. Rotate outfalls for duplicate analysis.

7. How do I set-up duplicates for BOD/CBOD and how often must they be run?
    a. To set-up duplicate BOD/CBOD samples, you will need to prepare identical dilution series for the duplicates. If the sample has four dilutions, then the duplicate will have the same four dilutions. Calculate the BOD/CBOD for each sample and use the average of the two concentrations for reporting purposes. You do not need to set-up additional blanks, seed series, or GGAs to analyze duplicate BODs or CBODs.

       The frequency of duplicates for BOD/CBOD is one duplicate sample per 20 samples (5% of samples) if citing 18th or 19th editions of *Standard Methods,* and once per week of analysis if citing the 20th or 21st editions. To simplify the process when using the 18th or 19th edition we recommend analyzing a duplicate monthly or every two months depending on the number of BODs typically analyzed a week. This way you don't have to keep a running count of BOD samples.

8. My facility uses continuous monitoring for determining pH and DO. What quality control am I required to perform for these tests?

a. Continuous or *in situ* monitoring requires daily calibration/verification of calibration. In addition, you will need to verify that the thermometer or thermistor is functioning properly once a year.

9. What is the purpose of quality control samples?
   a. **Reagent Blank (RB) or blank** – This is DI / lab water that has gone through exactly the same test process as the samples. Presence of the substance you are testing for in this QC sample indicates contamination in the reagents or the overall process. The concentration of the RB should be below the MQL. [See # 10]
   b. **Laboratory Fortified Blank (LFB) or "external source"** – This is a blank (DI / lab water) that has a known amount of analyte (substance you are testing for) added to it. Prepare it the same way you prepare a standard, but you must use a different source (a different lot number or another manufacturer) than the one used to prepare the calibration standards. It must go through the same process as the samples. Use it to check the reliability of the calibration standards and determine if you are loosing or gaining analyte during testing. High or low recovery indicates that the calibration standards may no longer be valid; that either the LFB or the standards were improperly prepared; the testing procedure is contaminating the samples; or analyte is being lost during the testing process due to leaks or some other problem. Note that the following will not have LFBs: pH, DO, TSS, TDS, TS, TVS, bacteriological tests, color, temperature, specific conductance, or turbidity.
   c. **Duplicates** – See question # 5.
   d. **Matrix Spike (MS) or Known Additions** – This is effluent (i.e., matrix) that has a known amount of analyte added to it (i.e., spiked). Prepare it the same way that you would prepare a LFB but use effluent instead of DI water. After it is spiked, the MS must go through the same process as the samples. If the recovery is higher or lower than the acceptable 80-120% (or method specified range) and all other QC is acceptable, it is an indication that something is present in the effluent that is causing the concentration to appear higher or lower than it is actually is. This is known as either a positive or a negative interferent. Note that the following will not have a MS: pH, DO, TSS, TDS, TS, TVS, bacteriological tests, BOD, CBOD, color, temperature, specific conductance, turbidity, or TRC.
   e. **Laboratory Control Sample (LCS)** – This is the same as a LFB. When possible, the analyst should not know the true value of the LCS. While a LFB is analyzed with each set of samples, a LCS is analyzed quarterly, semi-annually, or annually depending on how frequently your lab runs a given test. If you are running a LFB each day of testing for a given parameter, you do not need to test a LCS for that test. The purpose of this QC sample is to evaluate the overall testing procedure used by the laboratory.

10. What is a MQL and how do I determine it?

a. MQL stands for Minimum Quantitation Level. It is the lowest concentration of the substance you are testing for allowable in the reagent blank in your facility. The following tests will not have an MQL: BOD/CBOD, TSS, TDS, TVS, TS, DO, temperature, specific conductance, bacteriological tests, turbidity, TRC, and pH. Colorimetric and ISE methods may not have a MQL because for colorimetric methods, the RB is used to zero the instrument and for ISE, the sensitivity is too low at the low end of the curve to measure it.

Do the following to determine the MQL:

- Prepare a reagent blank [See # 9.a]
- Calibrate the instrument
- Take ten readings of the RB
- Using Excel or a scientific calculator, calculate the standard deviation of the ten readings. Be sure to include minus signs if they are part of the readings/concentrations recorded.
- Multiply the standard deviation by 10. This is the MQL.
- The MQL for any method should be lower than the QL for that method.

Each time you run a test, the RB must be less than the MQL for that test.

11. How often does the MQL have to be determined?

a. *Standard Methods* doesn't indicate how often the MQL must be determined. If the RB is routinely failing and you aren't able to figure out why, you should determine a new MQL for that test method.

12. How do I perform an initial demonstration of capability (IDC)?

a. *Note: DO, temperature, and bacteriological tests do not require IDCs*. Unless a method specifies how the IDC must be performed, follow these instructions.

- Each analyst/operator that performs a test procedure must prepare and analyze four ~~identical~~ samples of known concentration for that method.
  - Make the samples the same way as the LFB [See # 9.b] or purchase a sample of know concentration and divide it into 4 containers.
- Take the four samples through the entire testing process.
  - You will need a reagent blank [See # 9.a] if citing *Standard Methods 20th* or *21st Edition.* The concentration of the RB must be below 50% of the calculated Minimum Quantitation Level (MQL).
- Must recover:
  - 90-110 % of known concentration for 18th & 19th Ed. of *Standard Methods* for all parameters except BOD, which is 80-120%.

OR

- 80-120 % of known concentration for 20th & 21st Ed. of *Standard Methods*.

NOTE: pH must be +/- 0.1 S.U. of the known value instead of a percentage.

- If any of the four sample recoveries fail to meet the required percentage, the entire process must be repeated.
- Records must be maintained for each operator/analyst while they are conducting the test and for three years after they have stopped doing the testing. Include in the records:
  - Method number,
  - Preparation logs,
  - Benchsheets,
  - Calculations indicating % recovery,
  - Analyst initials with time and date of analysis,
  - Whether or not the IDC is acceptable.
- Each operator/analyst must do a new IDC when the equipment type changes.

13. I currently use HACH Pocket Colorimeter (Method 8167) to analyze TRC. Can I still use that method and what QC do I need to do?
    a. HACH Method 8167 is equivalent to *Standard Methods* 4500-Cl G. You must obtain a letter from HACH stating which edition of *Standard Methods* they are referencing and then perform the QC that is required by that specific edition.
       - You will need to perform an Initial Demonstration of Capability (IDC) [See #10] using a standard that you have prepared and divided into four beakers/containers. You may NOT use a Spec ✔ for IDC.
       - On each day of testing effluent samples, you will need to read the high and low Spec ✔ (This is your Laboratory Fortified Blank. HACH has calibrated the colorimeter for you at the factory so the Spec ✔ is from a source that is different from the standards used to calibrate the instrument.)
       - 18th & 19th ed. of *Standard Methods* require that one of every 20 samples tested be a duplicate sample; 20th & 21st ed. require that a duplicate be analyzed each day of testing or at least 5% of samples tested , whichever is more frequent.

14. My pH meter only allows a two-point calibration and Standard Methods calls for a three-point calibration. How can I meet the method requirement?
    a. Calibrate the meter using two pH buffers, usually 7 and 4 or 7 and 10~~, or 4 and 10~~. Then verify that the curve is valid over the desired range, typically pH 4 to pH 10, by reading a third buffer as a sample, the result of which must

be within +/- 0.1 SU of the actual buffer value. (The buffer used to verify the calibration acts as the third calibration point.) If the third buffer fails, recalibrate. You might try using the 7 and 4 buffers if you previously used 7 and 10 buffers or vice versa.